

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION

Unclassified

2a. SECURITY CLASSIFICATION AUTHORITY

ELECTED

JUN 20 1990

1b. RESTRICTIVE MARKINGS

3. DISTRIBUTION/AVAILABILITY OF REPORT

Approved for public release

AD-A223 227

S B

S B

S B

5. MONITORING ORGANIZATION REPORT NUMBER(S)

ARO 27017.1-M-S-A

6a. NAME OF PERFORMING ORGANIZATION

Corning Incorporated

6b. OFFICE SYMBOL
(If applicable)

7a. NAME OF MONITORING ORGANIZATION

U. S. Army Research Office

6c. ADDRESS (City, State, and ZIP Code)

SP DV 11
Corning, NY 14831

7b. ADDRESS (City, State, and ZIP Code)

P. O. Box 12211
Research Triangle Park, NC 27709-22118a. NAME OF FUNDING/SPONSORING
ORGANIZATION

U. S. Army Research Office

8b. OFFICE SYMBOL
(If applicable)

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER

DAAL03-89-C-0015

8c. ADDRESS (City, State, and ZIP Code)

P. O. Box 12211
Research Triangle Park, NC 27709-2211

10. SOURCE OF FUNDING NUMBERS

PROGRAM
ELEMENT NO.PROJECT
NO.TASK
NO.WORK UNIT
ACCESSION NO.

11. TITLE (Include Security Classification)

(U) Advanced Ceramic Armor Materials

12. PERSONAL AUTHOR(S)

S. L. Hagg, T. D. Ketcham, P. C. Merkel, L. S. Share

13a. TYPE OF REPORT

Final

13b. TIME COVERED

FROM 15 Aug '89 TO 31 Dec '89

14. DATE OF REPORT (Year, Month, Day)

5-11-90

15. PAGE COUNT

68

16. SUPPLEMENTARY NOTATION

The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

17. COSATI CODES

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)

FIELD	GROUP	SUB-GROUP

Ceramic, Armor, Toughened Alumina, Materials, Fabrication,
Ballistic Evaluation, Zirconia

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This report describes the research completed by Corning Incorporated under the U.S. Army Research Office contract #DAAL03-89-C-0015. The purpose of this program was to develop improved low-cost ceramic materials and processing technology for advanced armor systems, specifically for use in heavy vehicle armor to defeat long rod kinetic energy threats.

Several toughened alumina targets were fabricated and characterized. Ballistic evaluation was completed by the Southwest Research Institute.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT

 UNCLASSIFIED/UNLIMITED SAME AS RPT. DTIC USERS

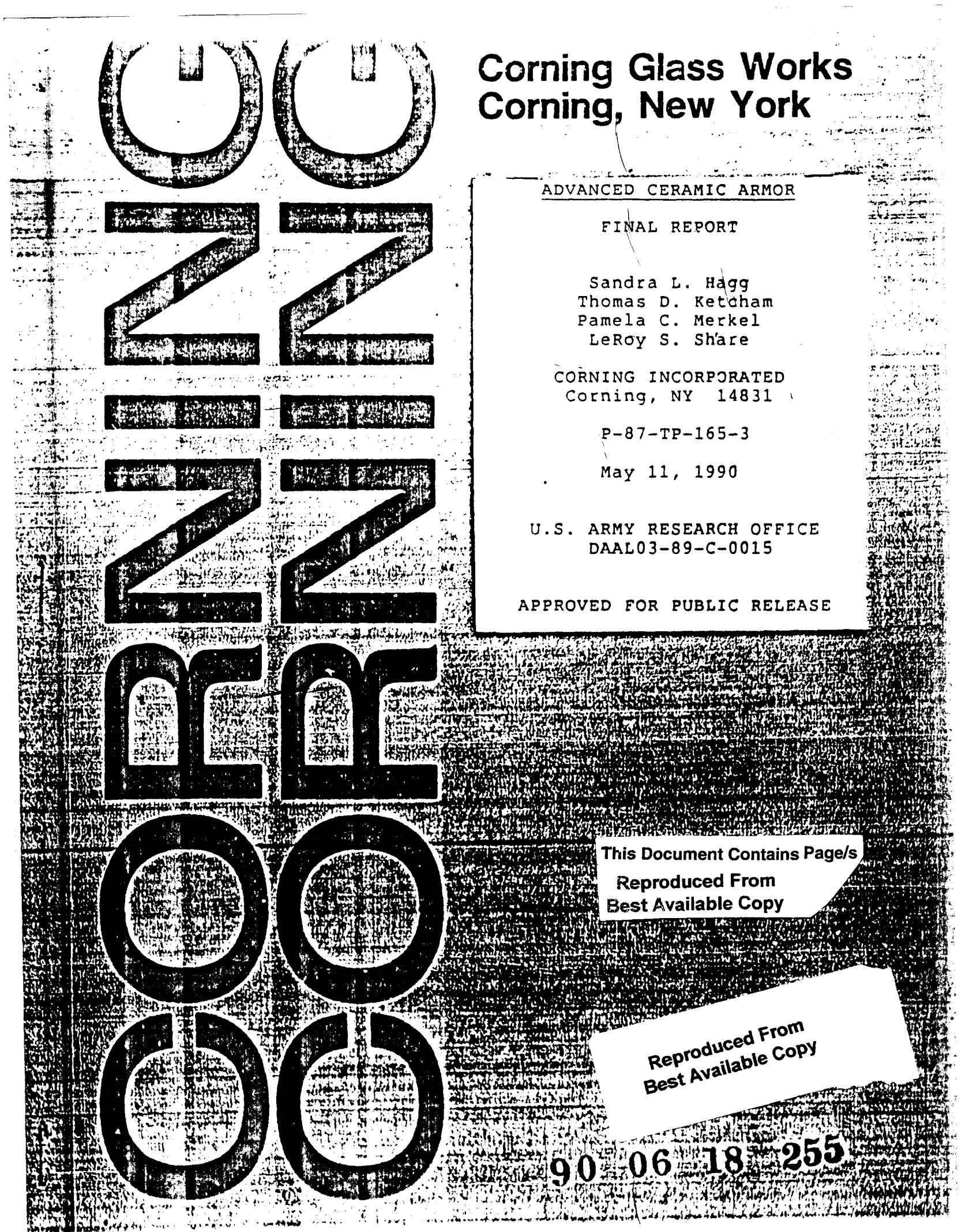
21. ABSTRACT SECURITY CLASSIFICATION

Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL

22b. TELEPHONE (Include Area Code)

22c. OFFICE SYMBOL



Corning Glass Works Corning, New York

ADVANCED CERAMIC ARMOR

FINAL REPORT

Sandra L. Hagg
Thomas D. Ketcham
Pamela C. Merkel
LeRoy S. Share

CORNING INCORPORATED
Corning, NY 14831

P-87-TP-165-3

May 11, 1990

U.S. ARMY RESEARCH OFFICE
DAAL03-89-C-0015

APPROVED FOR PUBLIC RELEASE

This Document Contains Page/s
Reproduced From
Best Available Copy

Reproduced From
Best Available Copy

90 06 18 255

ADVANCED CERAMIC ARMOR

FINAL REPORT

Sandra L. Hagg
Thomas D. Ketcham
Pamela C. Merkel
LeRoy S. Share

CORNING INCORPORATED
Corning, NY 14831

P-87-TP-165-3

May 11, 1990

U.S. ARMY RESEARCH OFFICE
DAAL03-89-C-0015

APPROVED FOR PUBLIC RELEASE

THE VIEW, OPINIONS, AND/OR FINDINGS CONTAINED IN THIS REPORT
ARE THOSE OF THE AUTHORS AND SHOULD NOT BE CONSTRUED AS AN
OFFICIAL DEPARTMENT OF THE ARMY POSITION, POLICY, OR
DECISION, UNLESS SO DESIGNATED BY OTHER DOCUMENTATION.

FORWARD

This report contains subject matter that has been placed under a secrecy order by the United States Department of Commerce, Patent and Trademark Office. See Appendix A.

Material in this document may be subject to ITAR restrictions prohibiting distribution to non-resident aliens in the United States and abroad.



Accession For	
NTIS GRA&I	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By _____	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
Forward	
1.0 Statement of the Problem Studied	1
2.0 Summary of the Results	1
2.1 Fabrication	1
2.2 Characterization	7
2.3 Ballistic Evaluation	7
2.4 Conclusions	7
3.0 Proposed Future Work	22
4.0 Publications and Technical Reports	24
5.0 Advanced Degrees Earned	25
6.0 Appendices	25

APPENDICES

A. United States Department of Commerce Secrecy Order	26
B. Ballistic Evaluation of ZTA and Zirconia	30
C. Corning Incorporated Facilities and Armor Experience	63

FIGURES

1. Hot Pressed Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGN-124, 2000 X Magnification	9
2. Hot Pressed Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGN-124, 10,000 X Magnification	10
3. Hot Pressed Zirconia Toughened Alumina SEM Micrograph of Fractured Section, AGN-124, 10,000 X Magnification	11
4. Sintered Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGN-124, 2000 X Magnification	12

TABLE OF CONTENTS

<u>FIGURES</u> (continued)	<u>PAGE</u>
5. Sintered Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGN-124, 10,000 X Magnification	13
6. Sintered Zirconia Toughened Alumina SEM Micrograph of Fractured Section, AGN-124, 10,000 X Magnification	14
7. Hot Pressed Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGA-124, 2000 X Magnification	15
8. Hot Pressed Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGA-124, 10,000 X Magnification	16
9. Hot Pressed Zirconia Toughened Alumina SEM Micrograph of Fractured Section, AGA-124, 10,000 X Magnification	17
10. Sintered Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGA-124, 2000 X Magnification	18
11. Sintered Zirconia Toughened Alumina SEM Micrograph of Polished Section, AGA-124, 10,000 X Magnification	19
12. Sintered Zirconia Toughened Alumina SEM Micrograph of Fractured Section, AGA-124, 10,000 X Magnification	20
<u>TABLES</u>	
1. Zirconia Toughened Alumina Batch Compositions	3
2. Properties of Starting Materials	4
3. Weight Loss and Shrinkage of Sintered Samples	5
4. Zirconia Toughened Alumina Mechanical Property Data	8
5. Ballistic Samples Supplied to Southwest Research Institute	21

1.0 STATEMENT OF THE PROBLEM STUDIED

This report describes the research completed by Corning Incorporated under the U.S. Army Research Office - DARPA contract #DAAL03-89-C-0015. The purpose of this program was to develop improved low-cost ceramic materials and processing technology for advanced armor systems, specifically for use in heavy vehicle armor, to defeat long rod kinetic energy threats.

Corning submitted a proposal for a one-year program in December 1987. The first phase was defined to provide samples of Exchange Reaction Hot Pressed (ERHP) materials, toughened alumina, fiber-reinforced glass matrix composites, and multilayer-gradient materials for ballistic testing. Fabrication and complete characterization of each material were included in our program. Southwest Research Institute was given responsibility for ballistic evaluation of all of the program participants' materials. Due to funding constraints, Corning was only funded for four months (September to December 1989). As a result, Corning limited the work scope to the fabrication and characterization of zirconia toughened alumina materials. Previous ballistic data generated at the University of Dayton Research Institute had demonstrated zirconia toughened alumina as a potential armor candidate (Appendix B). This material also was determined to have the highest potential for meeting the projected ultimate material cost requirements desired under this program (\$10/pound).

2.0 SUMMARY OF THE RESULTS

2.1 Fabrication

As stated earlier, sample fabrication was limited to sintered and hot pressed zirconia toughened alumina materials. Two sets of materials comprised of three compositions were studied. Two

compositions of Corning standard zirconia toughened alumina were hot pressed and sintered. The second material investigated was a lower firing temperature, Corning proprietary zirconia toughened alumina.

The first set of zirconia toughened alumina powder batches was prepared similarly for both sintered and hot pressed samples. Table 1 lists batch compositions. Commercially available powders were used for fabrication, and the data is listed in Table 2. No binder was used in any of the compositions. Batch components were weighed and placed into a 1000 ml Nalgene bottle with 664 grams of 10 mm diameter and 1836 grams of 15 mm diameter commercial ZrO_2 -3m% Y_2O_3 mixing balls with 500 mls of deionized water. The batches were then ball milled for 72 hours. After milling, each batch was dried and forced through a 40-mesh (openings/square inch) plastic screen.

The sintered samples were dry pressed using a 5 1/8-inch diameter metal mold at 1000 psi, then isostatically pressed in a vacuum sealed rubber bag at 28 Kpsi (193-MPa), with a two-minute hold. The green discs were fired in an electric furnace, in air, using the following schedule: heated 100°C/hr. (1550°C six-hour hold) and cooled 100°C/hr. to room temperature. Shrinkage and weight loss information on the sintered discs can be found in Table 3.

The dried powder batches for the hot pressed samples were individually loaded into a four-inch diameter graphite mold. The molds were lined with 0.005-inch thick graphite foil to facilitate sample removal. Each disc was hot pressed at six Kpsi (41.3-MPa) under vacuum to a maximum temperature of 1450°C with a one-hour hold. A graphite element, resistance heated Vacuum Industries hot press was used. All of the pressure was removed after the end of the hold. The samples were allowed to cool in the hot press under vacuum.

TABLE 1

ZIRCONIA TOUGHENED ALUMINA BATCH COMPOSITIONS

	<u>AGA</u>	<u>AGN</u>
Alumina (Al_2O_3)	78.4 w%	85.0 w%
Zirconia (ZrO_2)	20.8 w%	14.0 w%
Niobium Oxide (Nb_2O_5)	0.3 w%	none
Yttrium Oxide (Y_2O_3)	0.3 w%	0.5 w%
Tantalum Oxide (Ta_2O_5)	none	0.5 w%

TABLE 2
PROPERTIES OF STARTING MATERIALS

	<u>Average Particle Size (microns)</u>	<u>Surface Area (m²/gm)</u>
Alumina	0.55	7.3
Niobium Oxide	1.40	4.9
Zirconia	1.46	14.5
2 m% Yttria Stabilized Zirconia	1.28	14.3
3 m% Yttria Stabilized Zirconia	1.40	15.9
Tantalum Oxide	1.8	26.5

TABLE 3

WEIGHT LOSS AND SHRINKAGE OF SINTERED SAMPLES

	<u>% Weight Loss</u>	% Shrinkage (Diameter)	
		<u>After Isopressing</u>	<u>Total</u>
AGA	0.9	15.7	20.4
AGN	0.8	16.2	22.8
LDX	2.1	15.2	23.8

As mentioned earlier, the second set of samples fabricated contained a Corning proprietary material. Therefore, the composition will not be disclosed in this report. The low-temperature sintering zirconia toughened alumina material was wet mixed in lots of 250 grams and rolled for three days in 1000 ml Nalgene bottles containing 500 mls of distilled water, 1836 grams of 15 mm commercial ZrO_2 -3m% Y_2O_3 milling media, and 664 grams of 10 mm commercial ZrO_2 -3m% Y_2O_3 milling media. After wet mixing, two of the 250 gram batches were dried together in Vycor^R pie plates loosely covered with aluminum foil. Then the 500 grams of powder were crushed with a rubber spatula before dry rolling for one hour in a 1000 ml nalgene bottle containing 1500 grams of 15 mm commercial ZrO_2 -3m% Y_2O_3 milling media. When dry milling was completed, the material was forced through a very fine meshed nylon screen.

Two thicknesses of low-temperature sintering billets were made. One size contained 860 grams of powder, whereas the other size contained 1300 grams. The powder was pressed in a 5 1/8-inch diameter die on a hydraulic press to ten tons. The pressed billets were placed in 5 1/4-inch diameter rubber molds and cold isostatically pressed to 28 Kpsi (192-MPa) with a two-minute hold at that pressure. Sintering was accomplished in an electric glo-bar furnace, in air, with the billets placed on fine alumina granules (for movement during shrinking). The firing schedule was: 11 hours to 550°C (50°C/hour), 6-hour hold, 11 hours to 1100°C (50°C/hour), 3-hour hold, and 11 hours to room temperature (100°C/hour).

With sintering completed, all of the billets were finished to thicknesses which would yield the 10 and 15 gm/cm² areal densities for ballistic testing. The discs were ground, flat and parallel, with a 240-mesh surface finish.

2.2 Characterization

All of the zirconia toughened alumina materials fabricated were characterized using several techniques. Property data for the zirconia toughened alumina samples can be seen in Table 4. Density and porosity measurements were made using the Archimedes' water absorption method. Rockwell A Hardness measurements were done on the carbide scale using a brale diamond penetrator. Toughness was determined using the short bar Chevron Notch method, with a 0.563-inch by 0.325-inch by 0.375-inch bar. Young's Modulus, Shear Modulus, Poisson's Ratio, and the sound speed in the material were measured on the four-inch diameter ballistic samples using a non-destructive sonic technique. SEM micrographs were also taken of each of the fabricated materials. These photographs can be seen in Figures 1 through 12 and depict the light colored zirconia grains dispersed in the darker alumina phase for both hot pressed and sintezed materials.

2.3 Ballistic Evaluation

A total of fifteen four-inch diameter discs of zirconia toughened alumina were shipped to Southwest Research Institute in December 1989 for ballistic evaluation against a long rod penetrator ($L/D=10$). Discs ground to both $10\text{gm}/\text{cm}^2$ and $15\text{ gm}/\text{cm}^2$ areal densities were supplied. Sample code numbers are listed in Table 5. The testing was scheduled for April 23 through 27, 1990. Two representatives from Corning Incorporated were present at the testing.

2.4 Conclusions

The successful scale-up of the sintered zirconia toughened alumina samples was an impressive accomplishment completed under this contract. Previous research had been done on 0.5-inch diameter by 0.25-inch thick samples. The ballistic targets

TABLE 4

ZIRCONIA TOUGHENED ALUMINA MECHANICAL PROPERTY DATA

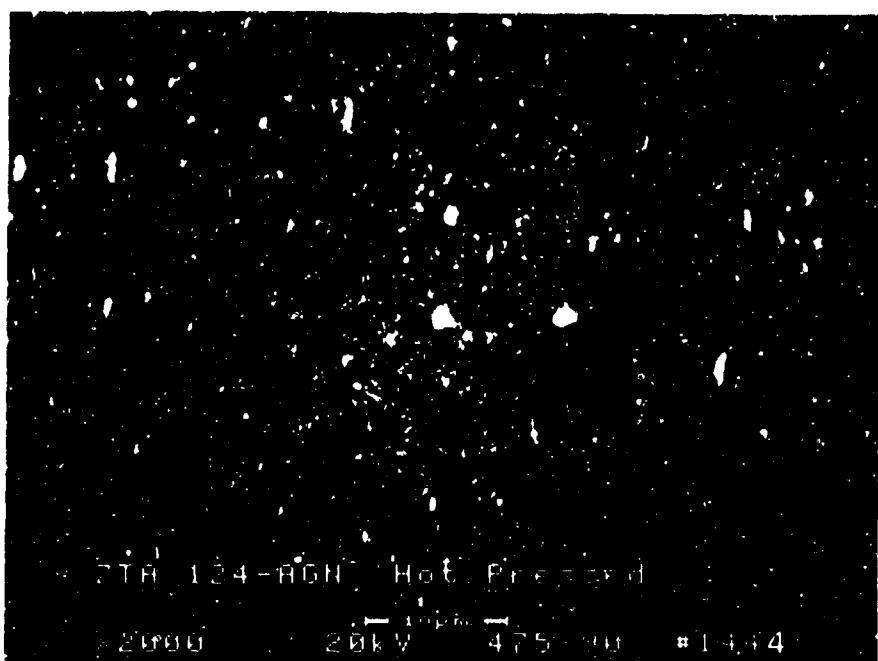
Processing Data		Density (gm/cc)	Closed Porosity (%)	Hardness Rockwell A	Toughness $K_{Ic}^{1/2}$ (MPa \cdot m $^{1/2}$)	Young's Modulus (Mpsi)	Shear Modulus (Mpsi)	Poisson's Ratio	Wave Velocity Long. (m/sec)	Shear (m/sec)
AGW	Hot Pressed	4.18	0.4	94.5	4.40	52.9	21.4	0.24	10140	5962
AGW	Sintered	4.17	0.7	92.8	4.72	53.6	21.6	0.24	10260	5976
AGA	Hot Pressed	4.25	1.0	90.2	5.14	52.8	21.3	0.24	10060	5874
AGA	Sintered	4.12	1.9	89.3	5.13	49.3	19.9	0.24	9806	5736
LDK	Sintered	4.13	---	89.8	----	42.3	17.1	0.24	9215	5389

FIGURE 1

Hot Pressed Zirconia Toughened Alumina

AGN-124

SEM Micrograph of Polished Section



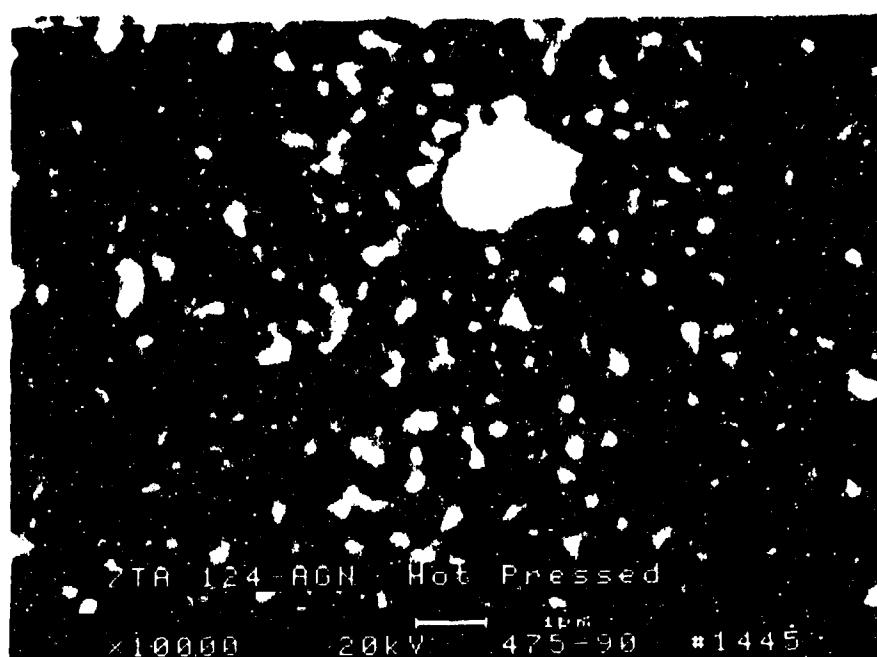
2000 X Magnification

FIGURE 2

Hot Pressed Zirconia Toughened Alumina

AGN-124

SEM Micrograph of Polished Section



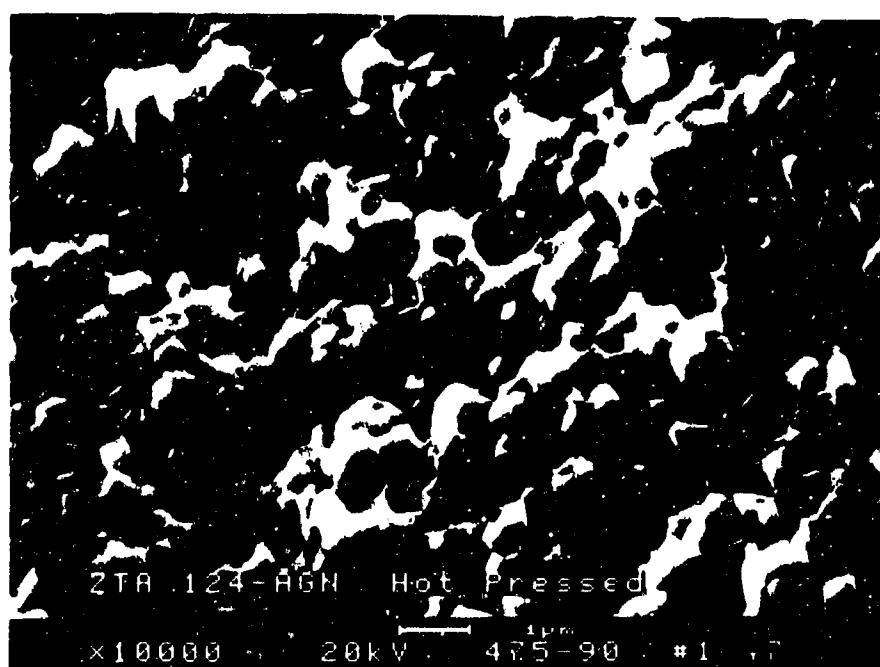
10,000 x Magnification

FIGURE 3

Hot Pressed Zirconia Toughened Alumina

AGN-124

SEM Micrograph of Fractured Section



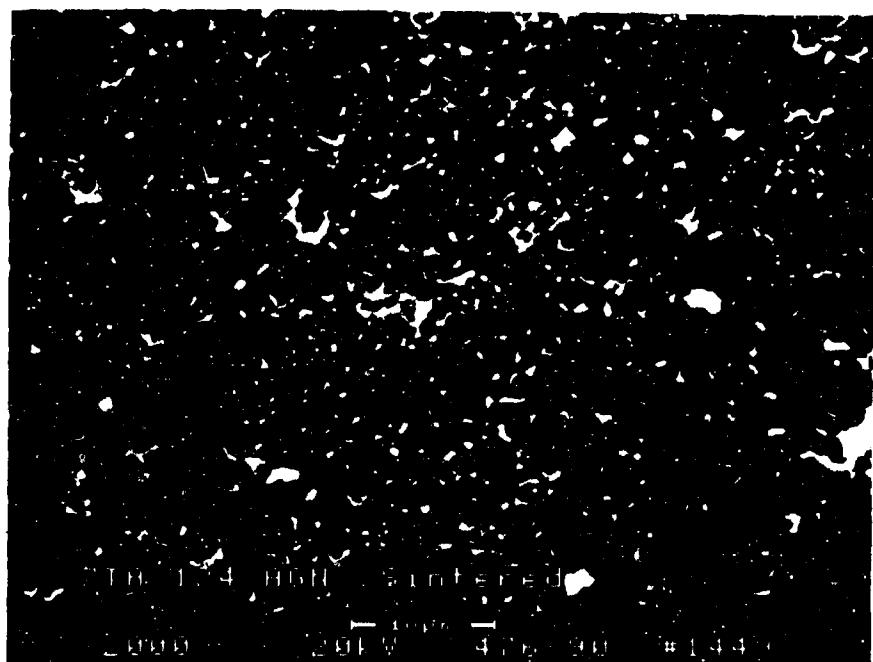
10,000 X Magnification

FIGURE 4

Sintered zirconia Toughened Alumina

AGN-124

SEM Micrograph of Polished Section



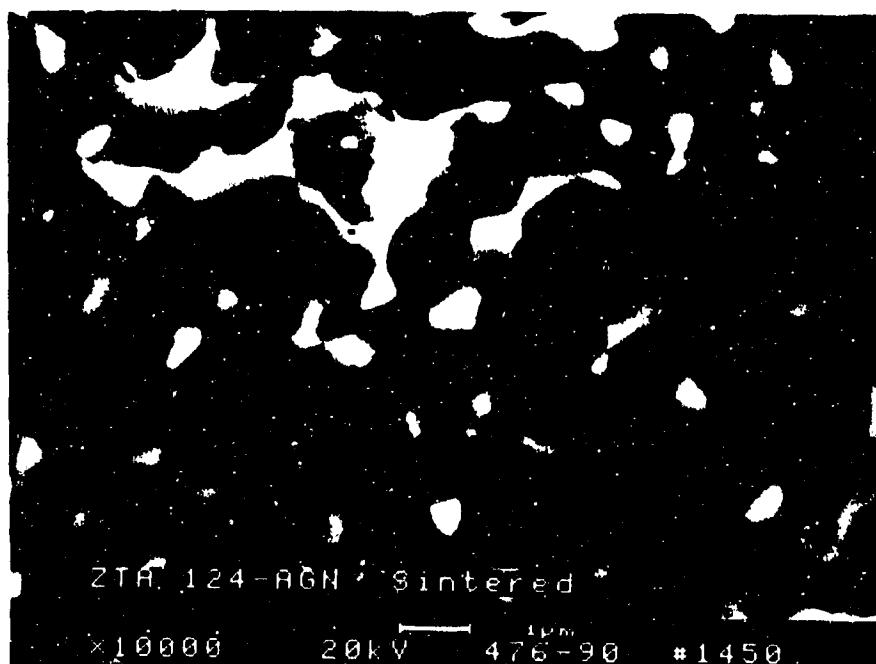
2000 X Magnification

FIGURE 5

Sintered Zirconia Toughened Alumina

AGN-124

SEM Micrograph of Polished Section



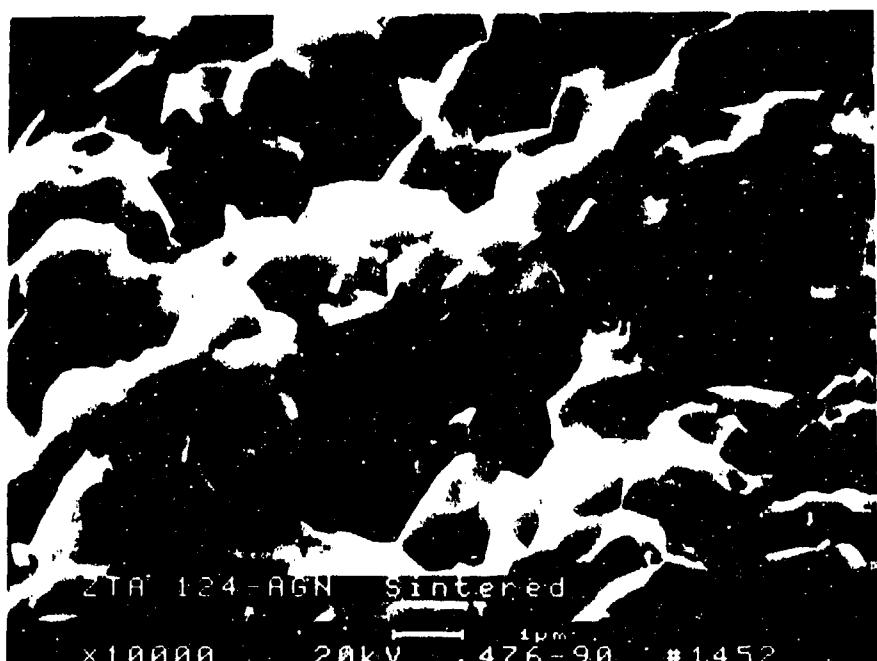
10,000 X Magnification

FIGURE 6

Sintered Zirconia Toughened Alumina

AGN-124

SEM Micrograph of Fractured Section



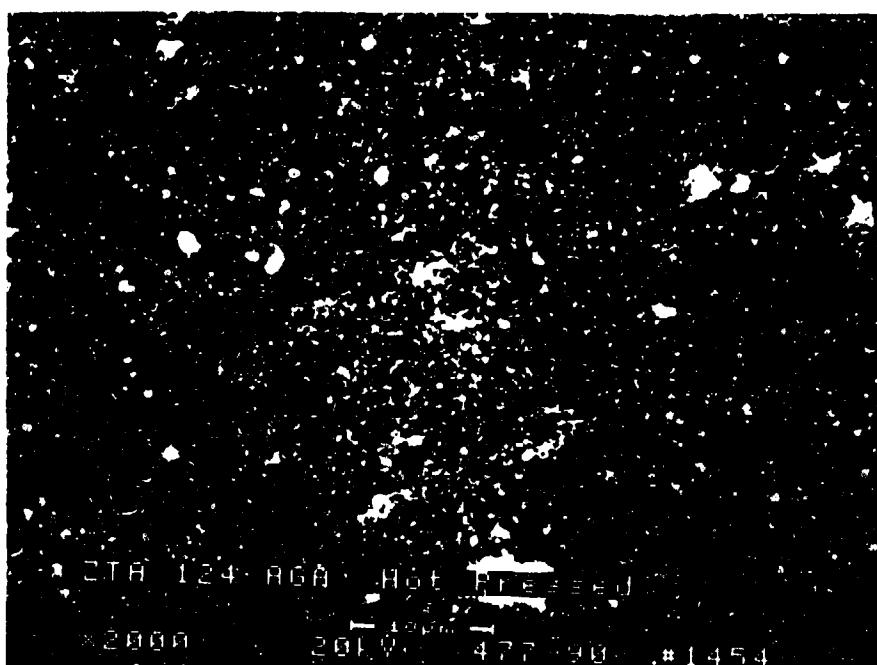
10,000 x Magnification

FIGURE 7

Hot Pressed Zirconia Toughened Alumina

AGA-124

SEM Micrograph of Polished Section



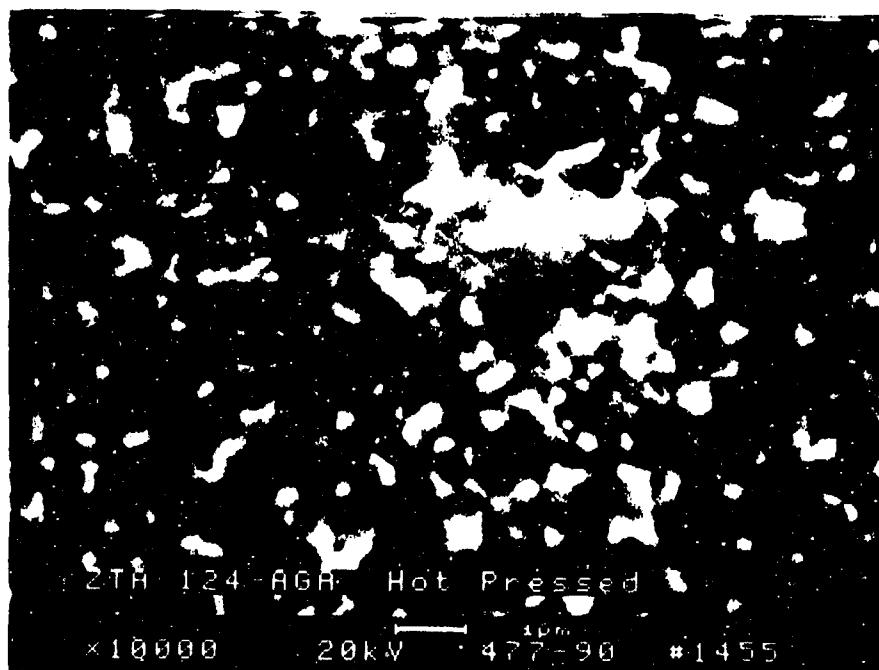
2000 X Magnification

FIGURE 8

Hot Pressed Zirconia Toughened Alumina

AGA-124

SEM Micrograph of Polished Section



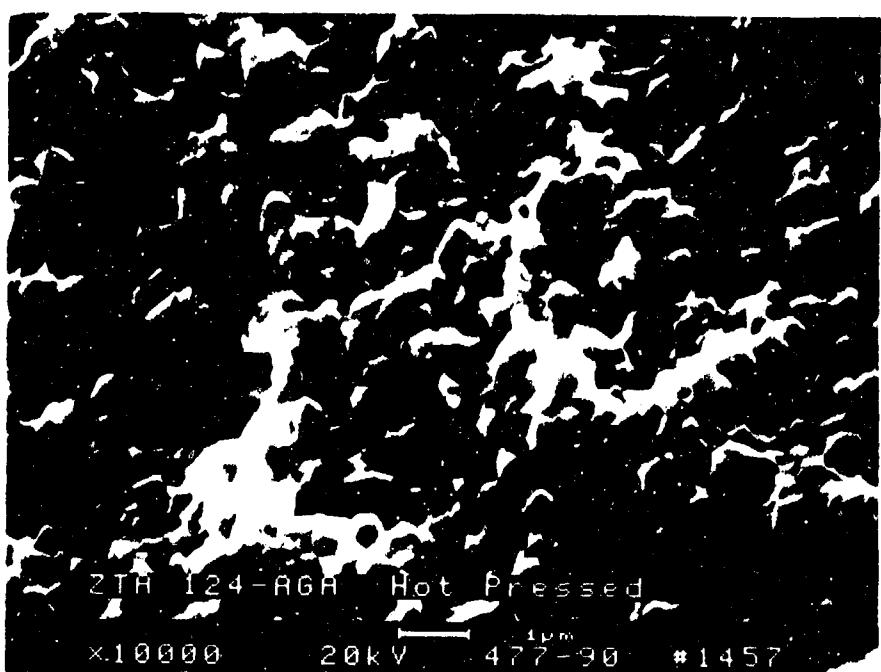
10,000 X Magnification

FIGURE 9

Hot Pressed Zirconia Toughened Alumina

AGA-124

SEM Micrograph of Fractured Section



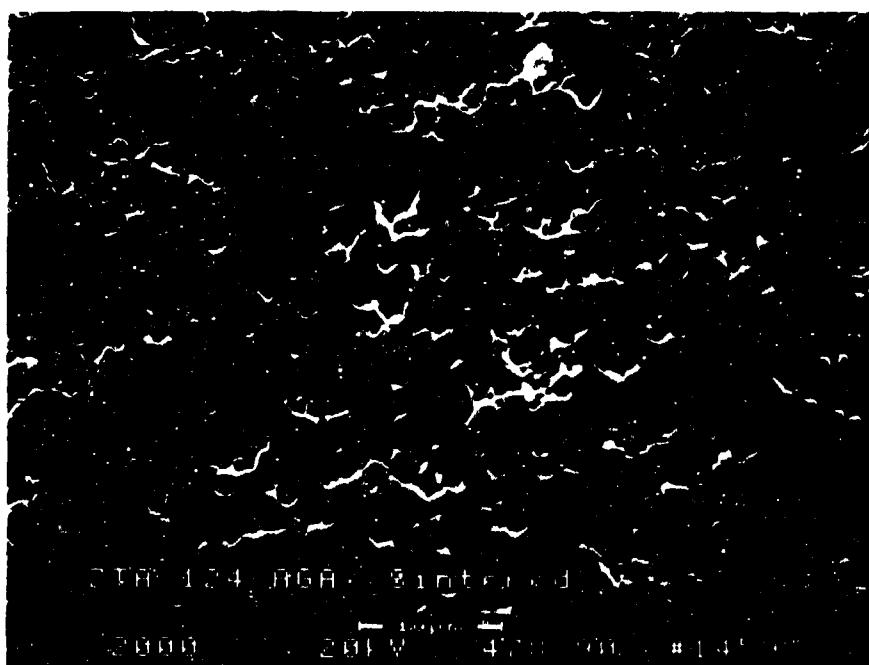
10,000 X Magnification

FIGURE 10

Sintered Zirconia Toughened Alumina

AGA-124

SEM Micrograph of Polished Section



2000 x Magnification

FIGURE 11

Sintered Zirconia Toughened Alumina

AGA-124

SEM Micrograph of Polished Section



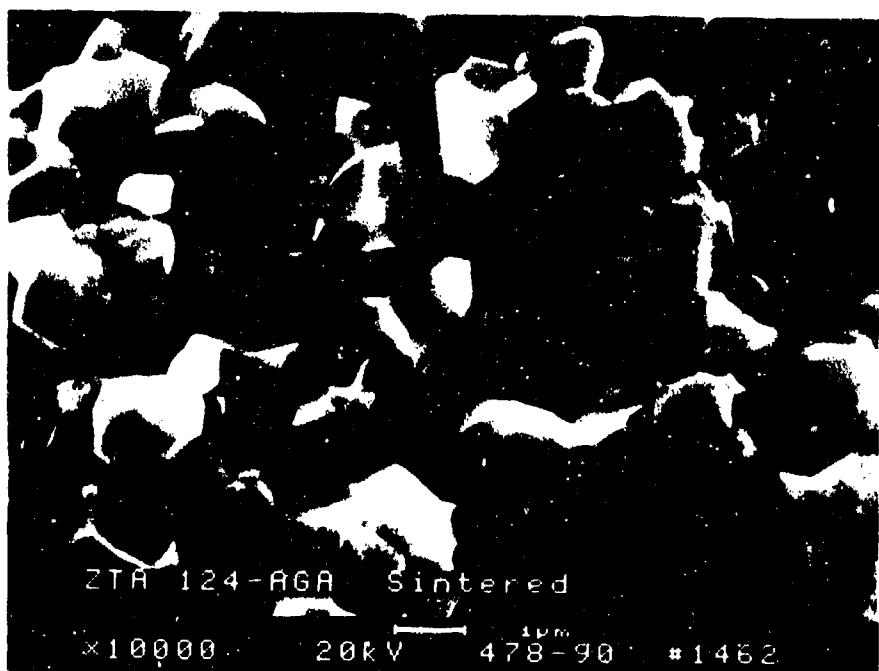
10,000 X Magnification

FIGURE 12

Sintered Zirconia Toughened Alumina

AGA-124

SEM Micrograph of Fractured Section



10,000 X Magnification

TABLE 5

BALLISTIC SAMPLES SUPPLIED TO SOUTHWEST RESEARCH INSTITUTE

<u>Sample Identification</u>	<u>Composition</u>	<u>Process Data</u>	<u>Areal Density</u>
7310-1			
7310-2	AGN	Hot Pressed	10 gm/cm ²
7310-3			
7310-4			
7310-5	AGN	Sintered	10 gm/cm ²
7310-6			
7310-10			
7310-11	AGA	Sintered	10 gm/cm ²
7310-19			
7310-13			
7310-14	LDX	Sintered	10 gm/cm ²
7310-15			
7310-16			
7310-17	LDX	Sintered	15 gm/cm ²
7310-18			

required for this contract were considerably larger: 4-inch diameter by 1.0-inch thick.

It was determined that adequate green strength could be achieved using these processing techniques without the use of a polymer binder system. This eliminated the need for a binder burnout step in the firing stage of processing.

As predicted, the compositions containing the highest weight percent alumina exhibited higher hardness and lower toughness values. Mechanical properties for the hot pressed samples were slightly superior to those of the sintered samples. The average grain size of the sintered material (one to two microns) was slightly larger than that of the hot pressed zirconia toughened alumina (less than one micron), largely due to the elevated fabrication temperature and longer hold time. It will be interesting to compare ballistic data for these materials.

Unfortunately, no ballistic data was available at the time this report was published. Therefore, no conclusions can be drawn as to the ballistic performance of sintered versus hot pressed zirconia toughened alumina against the long rod penetrator threat.

3.0 PROPOSED FUTURE WORK

All of the research completed under this contract involved hot pressed and sintered zirconia toughened alumina materials due to the short duration of the contract and funding constraints. It was believed that these materials had the highest potential for success against the specified threat at low cost. As discussed previously, Corning's original program also included the fabrication and characterization of Exchange Reaction Hot Pressed materials; fiber-reinforced ceramic matrix composites; and multilayered-gradient glass, glass-ceramic, and ceramic materials.

It is proposed that further research be conducted on each of the following material systems:

- Multilayer advanced armor materials consisting of a hard ceramic faceplate bonded to a graphite fiber-reinforced glass matrix composite backing.
- Multilayer advanced armor materials consisting of a hard faceplate with a fracture-resistant support, hot pressed in one step.
- Multilayer advanced armor materials consisting of laminated thin sheets, where at least two different compositions are used, with alternating layers of hard and ductile materials.
- Multilayer-gradient advanced armor materials consisting of laminated thin sheets where the composition is smoothly graded from the front face of the armor plate to the back.
- Low-cost sintered (without pressure) zirconia toughened alumina compositions.

The multilayer approach affords the opportunity to engineer properties into the armor body that would not be attainable in a single-phase, homogeneous, monolithic material. There could be as few as two layers, or there could be several thin layers built up into a thick body. One example proposed is the combination of a hard material which would act to break up a kinetic energy projectile, with a tough, fracture resistant material that would absorb some of the transferred energy and minimize collateral damage. Another possibility is a multilayer-gradient material system of laminated thin sheets, where composition is tailored to minimize or eliminate shock wave reflections. The entire

field of advanced ceramic materials including oxides, borides, nitrides, carbides, glasses, glass-ceramics, and graphite-glass composites could be utilized in layered structures.

It is also proposed that research be directed at new ceramic processing techniques which would lower the cost of advanced armor materials fabrication. Pressureless sintering of zirconia toughened alumina is one processing method being proposed to lower the cost of ceramic materials. The use of chopped graphite fibers and alternate consolidation techniques have the potential of lowering the cost of the graphite-glass composites.

This proposed work should be initiated without delay in order to maintain the continuity and critical skills that were assembled for its accomplishment. In addition, ballistic target fabrication and material characterization would be included in this effort. A third party jointly selected by Corning and DARPA would be enlisted to ballistically evaluate the materials developed. An attempt to link material properties with ballistic performance would also be part of the program.

4.0 PUBLICATIONS AND TECHNICAL REPORTS

No external publications or technical reports were published under this contract. A presentation on the program's progress was given at the DoD/DARPA review of Advanced Armor/Anti-Armor Materials sponsored by the Balance Technology Initiative on November 8 to 9, 1989, in Alexandria, Virginia.

5.0 ADVANCED DEGREES EARNED

No participating scientific personnel earned advanced degrees while employed under this contract.

6.0 APPENDICES

The following appendices are attached:

- Appendix A - United States Department of Commerce Secrecy Order.
- Appendix B - Ballistic Evaluation of ZTA and Zirconia (UDR-TR-89-58), by S. J. Bless and S. J. Manchak at the University of Dayton Research Institute.
- Appendix C - Corning Incorporated Facilities and Armor Experience.

APPENDIX A

United States Department of Commerce Secrecy Order



P-87-TP-165-3
Page 27
UNITED STATES DEPARTMENT OF COMMERCE
Patent and Trademark Office
ASSISTANT SECRETARY AND COMMISSIONER
OF PATENTS AND TRADEMARKS
Washington, D.C. 20231

[Serial No.] 07/402,515 Filing Date 09/05/89 First Named Applicant IE ANGEL IS Attorneys Docket No. 7 13410

CLINTON S. JANES, JR.
CORNING INCORPORATED - SP-FR-2-12
CORNING, NY 14831

Sponsoring agency & Address:
Mr. Anthony Lane
U.S. Army Patents, Copyrights
and Trademarks Division
Office of the Judge Advocate General
5611 Columbia Pike, Room 332A
Falls Church, VA 22041-5013
(703) 756-2434

Examiner

MARGARET H. P.
Art Unit

+18 Date Mailed

04/03/90

SECRECY ORDER

(Title 35, United State Code, sections 181-188 (1952))

NOTICE: To the applicant(s) above named; his, her or their heirs; and any and all of the assignees, licensees, attorneys and agents, hereinafter designated principals:

You are hereby notified that the above-identified patent application has been found to contain subject matter the unauthorized disclosure of which would be detrimental to the national security, and you are ordered to keep the subject matter secret (as required by 35 U.S.C. 181) and you are further ordered NOT TO PUBLISH OR DISCLOSE the subject matter to any person except as specifically authorized by the Commissioner of Patents and Trademarks.

Any other patent application already filed or hereafter filed in the U.S. or any foreign country which contains any significant part of the subject matter of the above-identified patent application falls within the scope of this Order. If such other patent application is not under a Secrecy Order imposed by the U.S. Patent and Trademark Office, it and the common subject matter immediately must be brought to the attention of the Director, Group 220, Attn: Licensing and Review, U.S. Patent and Trademark Office, Washington, D.C. 20231 as soon as possible.

Publication or disclosure of the subject matter of the above-identified patent application, except as authorized herein or subsequently by the Commissioner of Patent and Trademarks, may subject the person publishing or disclosing the subject matter to the penalties of 35 U.S.C. 182, 185 and 186 (1952).

Since the disclosure of the subject matter of the above-identified application would be detrimental to the national security, the subject matter must be safeguarded under conditions that will provide adequate protection and prevent access by unauthorized persons. When copies of the subject matter are no longer needed, they should be destroyed by any method that will prevent disclosure of the contents or reconstruction of the document.

This order should not be construed in any way to mean that the Government has adopted or contemplates adoption of the invention disclosed in this application and it is not any indication of the value of such invention.

Please contact the above sponsoring agency for information concerning the imposition of the Secrecy Order and its status. Contact the Licensing and Review Division of the Patent and Trademark Office (703/557-4949) for information regarding the examination of this patent application.

Charles T. Jordan
for Director, Special Laws Administration



P-87-TP-165-3
Page 29
UNITED STATES DEPARTMENT OF COMMERCE
Patent and Trademark Office
ASSISTANT SECRETARY AND COMMISSIONER
OF PATENTS AND TRADEMARKS
Washington, D.C. 20231

Serial No.	Filing Date	First Named Applicant	Attorneys Docket No.
07/402,515	09/05/89	DE ANGELIS	T 13410

CLINTON S. JAMES, JR.
CORNING INCORPORATED -SP-FR-2-12
CORNING, NY 14831

PERMIT A

Examiner

MARSHALL, R.

Art Unit

116

Date Mailed

04/07/90

An order of secrecy having been issued in the above-entitled application by the Commissioner of Patents and Trademarks, the principals as designated in said order are authorized to disclose the subject matter to any person of the classes hereinafter specified if such person is known to the principal disclosing to be concerned directly in an official capacity with the subject matter, provided that all reasonable safeguards are taken to otherwise protect the invention from unauthorized disclosure. The specified classes are:

- (a) Any officer or employee of any department, independent agency, or bureau of the Government of the United States
- (b) Any person designated specifically by the head of any department, independent agency or bureau of the Government of the United States, or by his duly authorized subordinate, as a proper individual to receive the disclosure of the above indicated application.

The principals under the secrecy order are further authorized to disclose the subject matter of this application to the minimum necessary number of persons of known loyalty and discretion, employed by or working with the principals or their licensees and whose duties involve cooperation in the development, manufacture or use of the subject matter by or for the Government of the United States, provided such persons are advised of the issuance of the secrecy order.

The provisions of this permit do not in any way lessen responsibility for the security of the subject matter as imposed by any Government contract or the provisions of the existing laws relating to espionage and national security.

for Charles T. Jordan
Director, Special Laws Administration

APPENDIX B

Ballistic Evaluation of ZTA and Zirconia

BALLISTIC EVALUATION OF ZTA AND ZIRCONIA

PURCHASE ORDER NO. ET-13492

BY:

S. J. HANCHAK AND S.J. HANCHAK

JULY 1989

PREPARED FOR:

**AMERICAN GLASS WORKS
RESEARCH & DEVELOPMENT DIVISION
ALBION, NY 14831**



University of Dayton

*Reproduced From
Best Available Copy*

TABLE OF CONTENTS

<u>Section</u>		<u>Page</u>
1	BACKGROUND	1
2	CORNING MATERIALS	5
3	TESTS CONDUCTED	6
4	CONCLUSIONS AND RECOMMENDATIONS	10
5	ACKNOWLEDGEMENTS	11
	REFERENCES	12
	APPENDIX	13

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Sketch of Geometry Used in Screening Experiments from Reference 1. The Dimensions Used here are for the LRP Penetrator.	2
2	Possible Outcomes of Screening Experiments.	3
3	Recovered Piece of Ceramic from Shot 1-0071 Showing Filaments of Metal (Tungsten) Coating Fragment. Width of Specimen is 19 mm (Measured Along Long Edge).	8
4	Comparison of Corning Materials with Other Ceramics Tested in Reference 1.	9

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	TESTED TILES	5
2	RESULTS OF TESTS	7

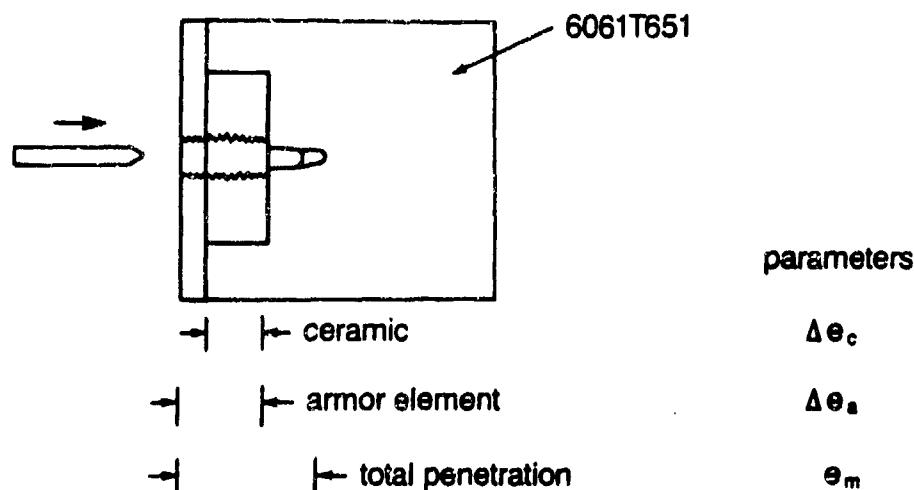
SECTION 1
BACKGROUND

The University of Dayton developed a test technique for screening ceramics during its effort conducted under the DuPont Armor Program (Reference 1). In order to evaluate ceramics, a screening technique is required whereby the performance of ceramics may be quantitatively compared. The screening technique should be sensitive to small changes in performance, since we are often trying to identify optimum ceramics. The screening technique should not require very many tests, since experimental ceramic compositions are often relatively expensive.

Ballistic limits are often used for screening test measurements. However, in the case of ceramic evaluation there are three disadvantages to ballistic limit measurements: (1) ballistic limits can be substantially increased by tuning the substrate to the ceramic element, but in the case of material evaluation, it is not known in advance how to best match the substrate to the ceramic; (2) ballistic limits are difficult to measure precisely, and change only slowly with material properties; and (3) precise determination of a ballistic limit requires at least six tiles, which may be very expensive.

The generic screening test geometry developed by the University and used in this program is sketched in Figure 1. There is a ceramic tile placed over a very thick aluminum substrate. The ceramic is confined on the sides by aluminum and usually on the impact side by a cover plate. The projectile usually overmatches the tile, so much of the projectile ends up in the substrate. Figure 2 illustrates the various possible outcomes of a test. Case 2 is the usual outcome. Measurement of W_R (equal to the residual penetration times the density) is taken as a characteristic of the ceramic. In most cases, W_R is a linear function of the ceramic areal density, W_C . Thus, $\Delta W_R / \Delta W_C$ is a material property of the ceramic which expresses its resistance to penetration. This technique of screening ceramics was

Screening Geometry



projectile	tile width (mm)	CP Mat'l	CP T (mm)
.5APDS	45	7017	10.3
.5LRP	75	6061T651	12.7

Figure 1. Sketch of Geometry Used in Screening Experiments from Reference 1. The Dimensions Used here are for the LRP Penetrator.

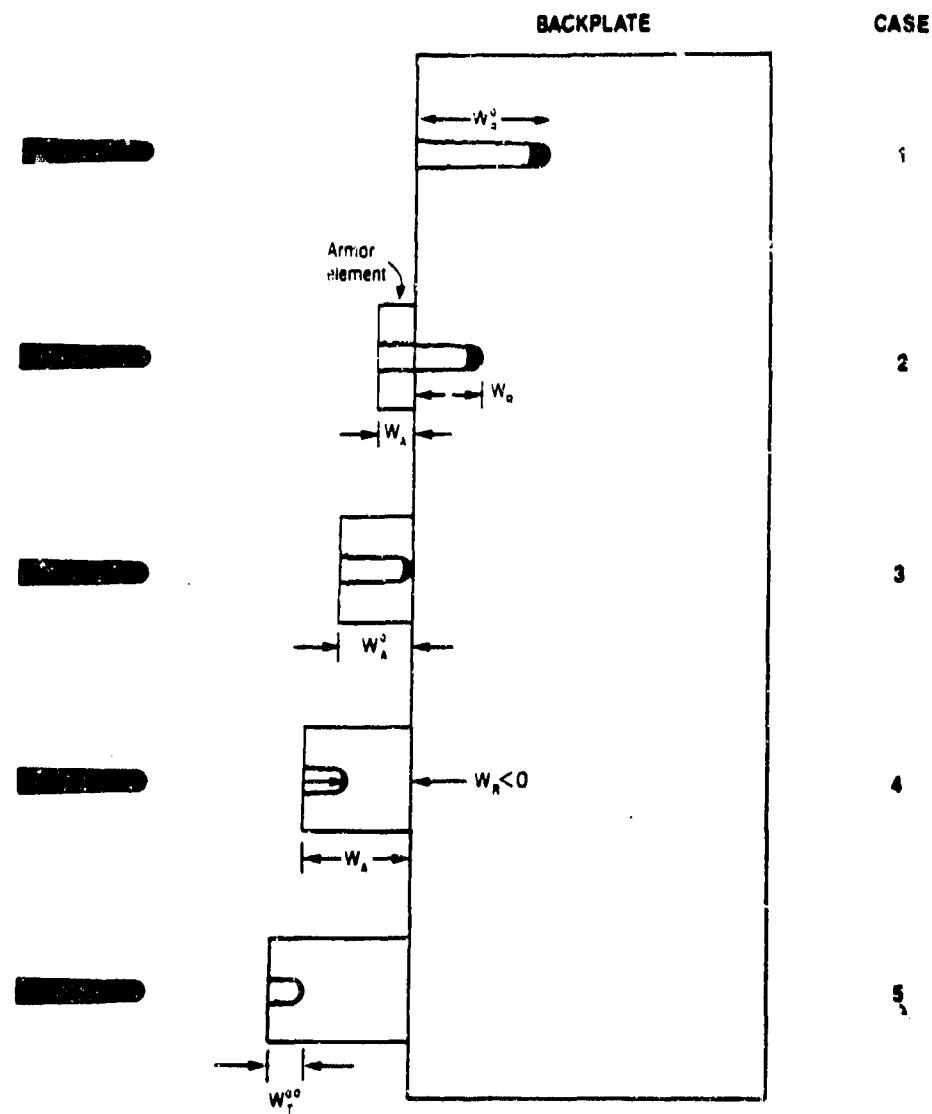


Figure 2. Possible Outcomes of Screening Experiments.

first described in Reference 2. The Differential Efficiency (DEF) is equal to $\Delta W_R / \Delta W_C$. It can be computed from

$$DEF = \frac{W_R^0 - W_R}{W_C} \quad (1)$$

(See Figure 2 for W_R^0 definition).

In order to save expense on materials and shooting expendables during screening in Reference 1, experiments were conducted half scale. Thus, the projectiles were half-scale versions of the two specified full-scale (30 mm) threats: Armor Piercing Discarding Sabot (APDS) and Long Rod Penetrator (LRP). The mass of the projectiles was 1/8 the full-scale mass. According to accepted principles, when experiments are carried out half-scale, but with full impact velocity and full-scale materials, then at corresponding locations and times, velocities, and stresses are the same in the half-scale test as they would be in a full-scale test.

The testing reported herein was conducted with a half-scale version of the DARPA 30 mm LRP threat. In this report we are not going to divulge the threat dimensions because the data in Reference 1, to which we want to refer, are CLASSIFIED when combined with a description of the projectile.

SECTION 2
CORNING MATERIALS

Corning supplied tiles denoted ZTA (Zirconia Toughened Alumina) and ZrO_2 . Table 1 lists the tiles that were tested. The density measurements were carried out by the University.

TABLE 1. TESTED TILES

<u>Tested</u>	<u>Ceramic</u>	Width (mm)	Thickness (mm)	Density (g/cm ²)
1-0071	ZTA	78.6	30.7	4.28
1-0072	ZTA	78.5	30.1	4.27
1-0073	ZrO_2	76.1	25.9	5.97
1-0074	ZrO_2	76.1	25.6	5.99

SECTION 3 TESTS CONDUCTED

The 20 mm Range used previously in Reference 1 was not set up at the University when Corning requested us to conduct the present program. However, a new and more sophisticated range was available, featuring a 30 mm smooth bore gun and a larger target tank. It was decided to set up the program on this new range because the expense per shot would be less (due to fewer failures). This process involved developing a new sabot and carrying out test shots on HRC35 4340 steel. The first test shot, 1-0070 was perfect, so subsequent shots were conducted against the targets of Table 1.

The results of the testing are summarized in Table 2. In the Appendix to this report are pictures of the coverplates, the appearance of the targets when the coverplate was first removed, and the appearance of the target when loose debris was removed. As necessary, substrates were thin sectioned and x-rayed to determine residual penetration. Prints of the residual penetration x-rays are also included in the Appendix.

The two zirconia tiles produced very similar results. However, there was an astonishing discrepancy between the two ZTA test results. In one case, Shot 1-0072, the rod failed to reach the substrate. The residual penetration was due to indents; there was no tungsten embedded in the aluminum. In the other ZTA shot, a substantial fraction of the penetrator passed into the substrate. In the successful shot the tile remaining in the cavity was extremely competent. In all other shots, it was relatively easy to remove material from the cavity. The discrepancy in the ZTA targets is larger than we have observed in any other material. We have no explanation.

Note that when there is no or very little tungsten in the substrate, as in Test 1-0072, the test results are less precise. It is quite likely that the tile thickness could be reduced

without more penetration resulting, because the rod was essentially fully consumed in the ceramic. Thus, the performance of the good ZTA target could probably have been even better if the tile were thinner.

There was an unusual clue relating to penetration mechanisms. In Shot 1-0071 an intact lower corner of the tile was recovered. The fracture was parallel to the side faces of the tile, as one would expect from an early spall failure. This recovered piece is shown in Figure 3. There are "cloisonné" like silver streaks on the ceramic. We hypothesize that these are due to tungsten that extruded into the radial cracks that existed during the penetration, which cracks did not extend into the decoupled fragment that we recovered.

Data from Corning tests are compared to other ceramics that were tested by the same technique as reported in Reference 1. The comparison is shown in Figure 4. It can be seen that the datum for HRC35 steel is in excellent agreement with the previous data. (The datum for Canasite was obtained with Corning donated material in Reference 1.) The data for zirconia indicate that this material is not a competitive ceramic. The best ZTA tile exhibited very competitive behavior.

Note that the HRC35 steel tested here is a little harder than thick sections of RHA, which are typically HRC26 to HRC30. Thus, e_m values cannot be estimated by ratioing to the steel data. HRC35 steel is, however, the hardness designated in the DARPA KE programs to represent future armor targets.

TABLE 2. RESULTS OF TESTS

Shot	Velocity (km/s)	Yaw (°)	Target	Penetration Below Tile (mm)
1-0070	1.38	0.5	Steel	37.3
1-0071	1.38	2.9	ZTA	52.4
1-0072	1.39	1.6	ZTA	0.5
1-0073	1.37	3.8	Zirconia	46.0
1-0074	1.39	3.6	Zirconia	52.2



Figure 3. Recovered Piece of Ceramic from Shot 1-0071 Showing Filaments of Metal (Tungsten) Coating Fragment. Width of Specimen is 19 mm (Measured Along Long Edge).

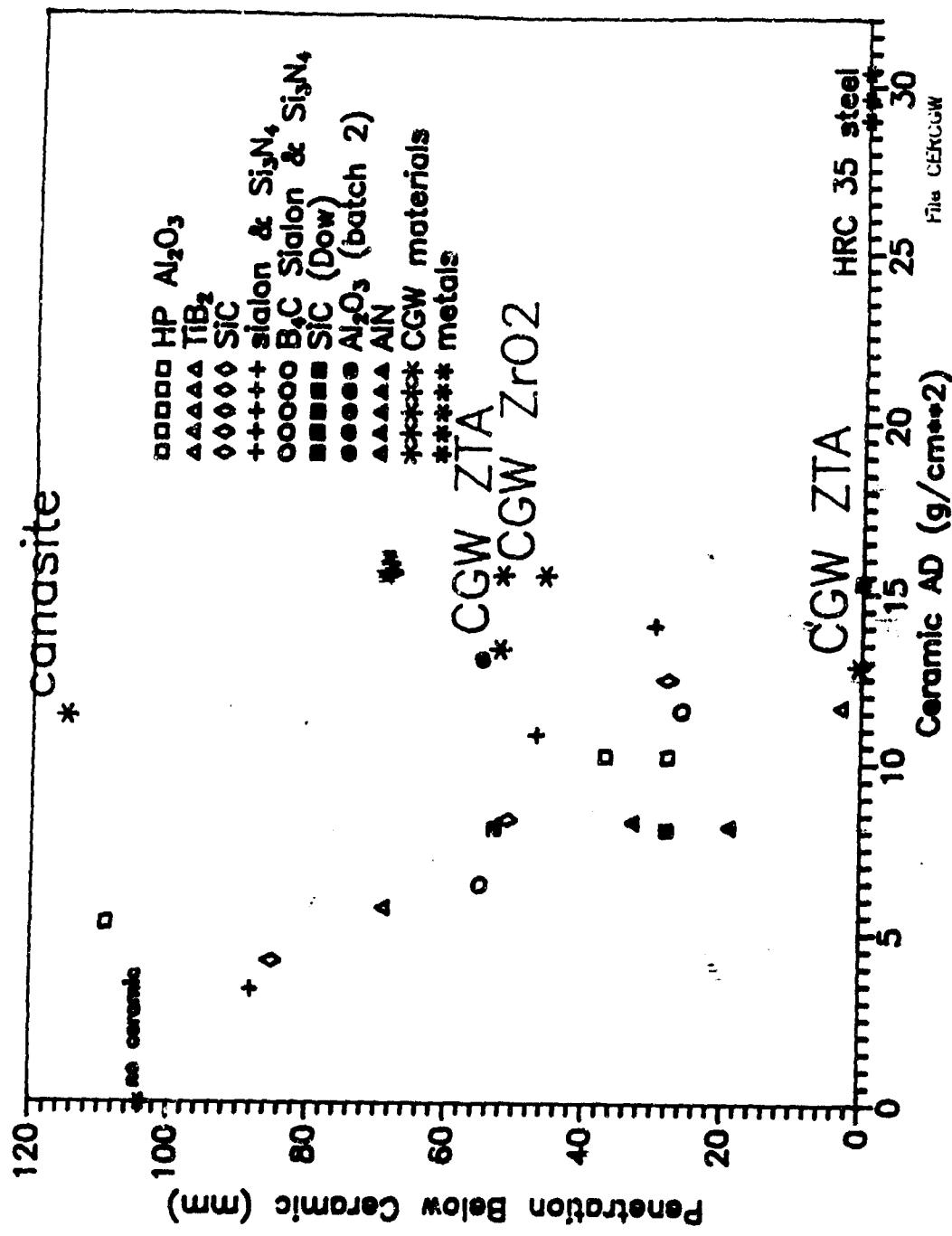


Figure 4. Comparison of Corning Materials with Other Ceramics Tested in Reference 1.

SECTION 4
CONCLUSIONS AND RECOMMENDATIONS

Zirconia does not appear to be a reasonable choice for an armor ceramic. The tiles that we tested were twice as heavy as other ceramics for the same level of protection.

ZTA seems very promising. There was an unaccounted for dispersion in test results with this material. However, the best ZTA was competitive with the best armor ceramics.

More experiments with ZTA should be conducted to determine the reason why one specimen performed so outstandingly. Good performing ZTA tiles should either be tested at 9 g/cm² areal density (for comparison with the best conventional ceramics), or they should be tested with longer rods so that some tungsten penetrates into the substrate. Corning should conduct a study of the microstructure of the impacted ZTA targets, paying particular attention to failure modes and grain boundary phases.

SECTION 5
ACKNOWLEDGEMENTS

Targets were prepared by Dr. N.S. Brar of the Impact Physics Laboratory. Sandra Hagg from Corning Glass Works assisted in target deconstruction and interpretation of results.

REFERENCES

1. S.J. Bless, J. Gonzalez, M. Alme, N.S. Brar, and C. Tarry, "Study to Design Ceramic Armor," 6th TACOM Conference on Light Armor Vehicles, Monterey, CA, March 1989.
2. Z. Rosenberg, S. Bless, Y. Yeshurun, and K. Okajima, "A New Definition of Ballistic Efficiency of Brittle Materials Based on the Use of Thick Backing Plates," Impact 87 Conference, Bremen, FRG, May 1987.

APPENDIX



Figure A1. Coverplate from Shot 1-0071.



Figure A2. Appearance of Target when Coverplate was Removed from Shot 1-0071.



Figure A3. Appearance of Target 1-0071 when Loose Debris was Removed.



Figure A4. Coverplate from Shot 1-0072.



Figure A5. Appearance of Target when Coverplate was Removed from Shot 1-0072.



Figure A6. Appearance of Target 1-0072 when Loose Debris was Removed.

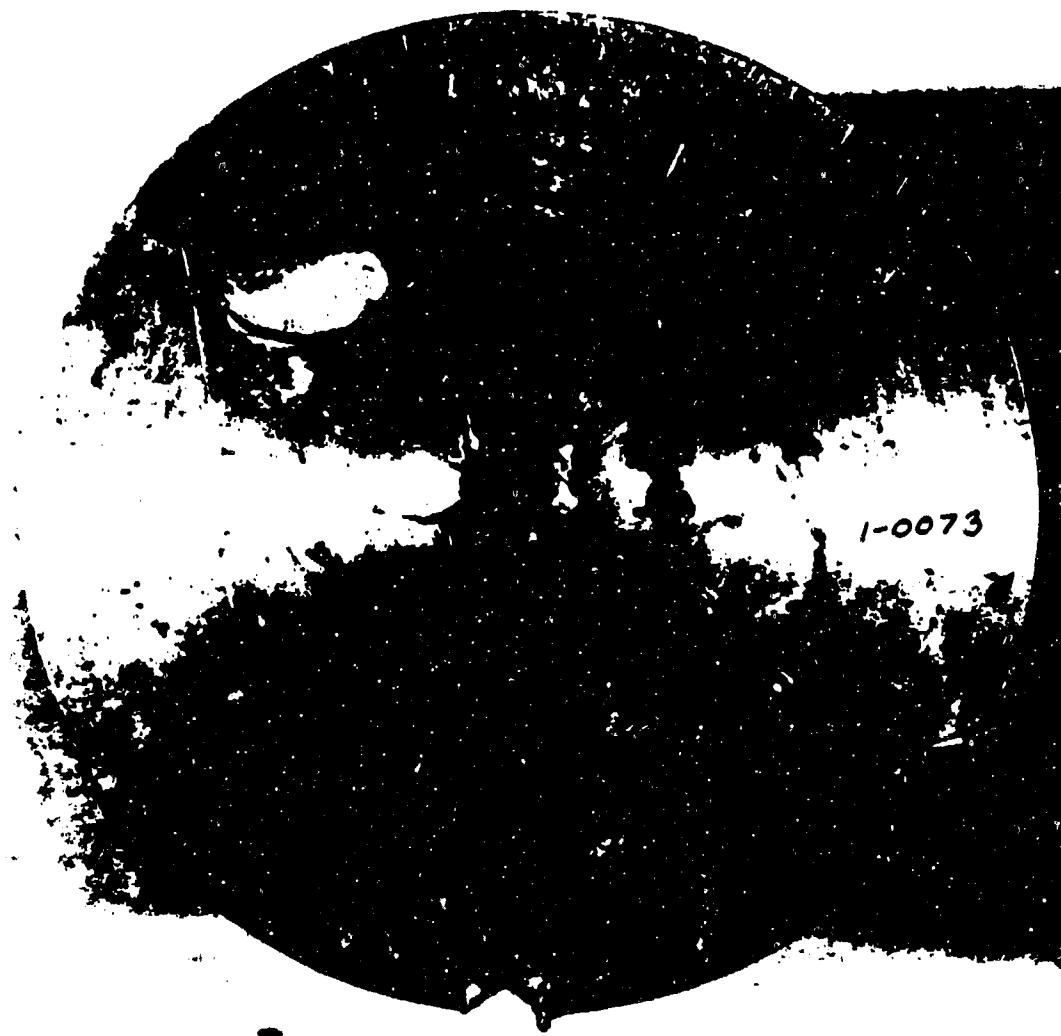


Figure A7. Coverplate from Shot 1-0073.

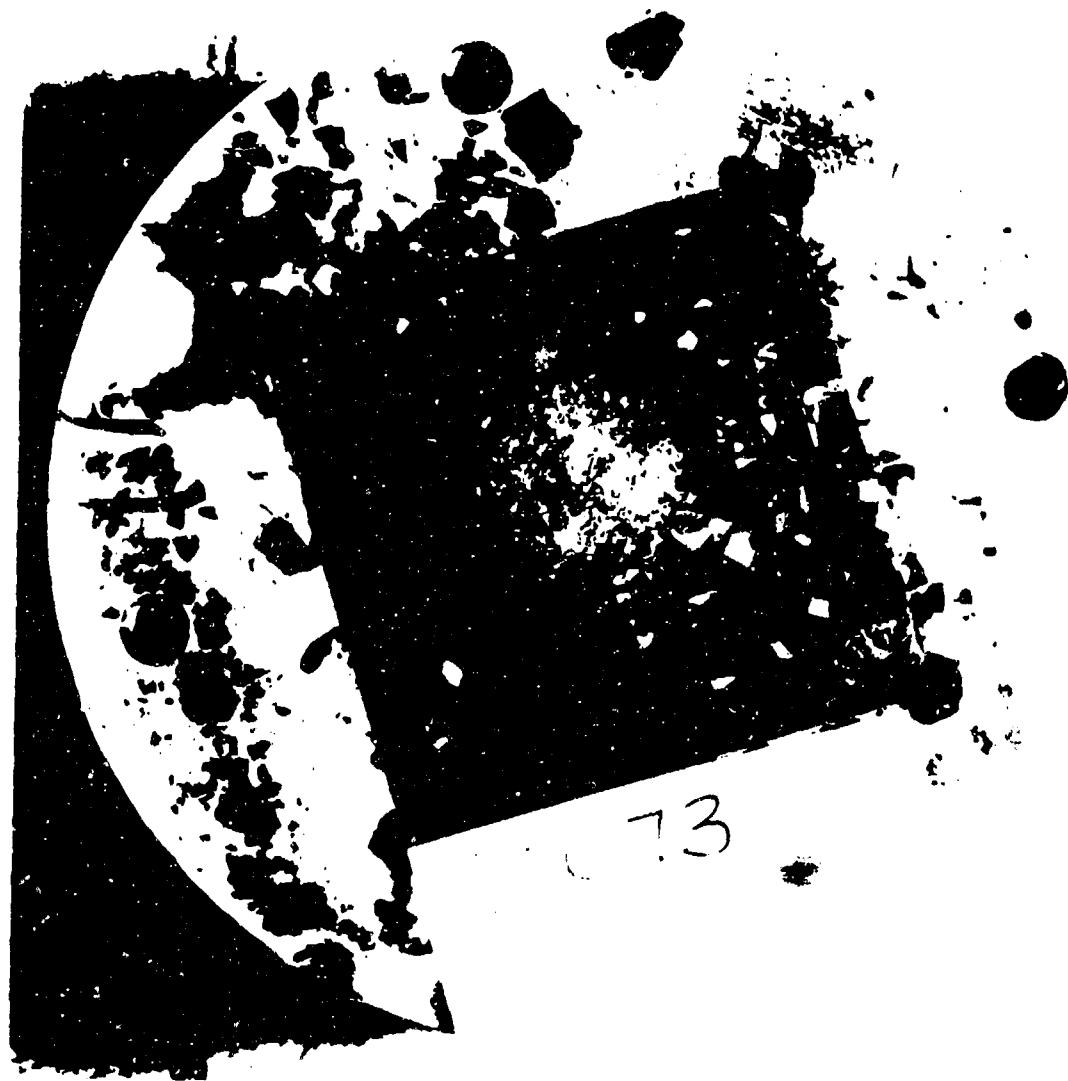


Figure A8. Appearance of Target when Coverplate was Removed from Shot 1-0073.

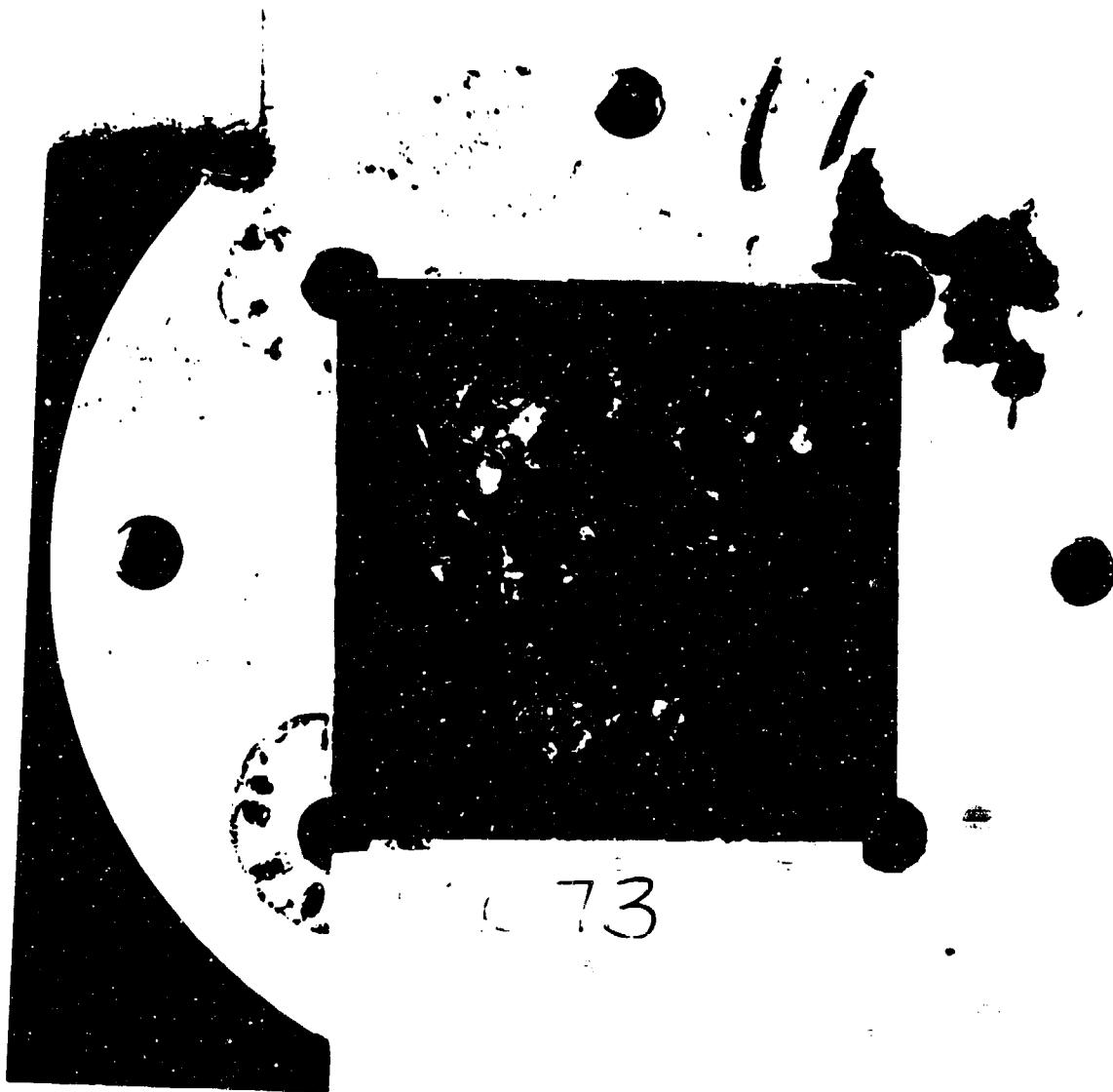


Figure A9. Appearance of Target 1-0073 when Loose Debris was Removed.



Figure A10. Coverplate from Shot 1-0074.



Figure All. Appearance of Target 1-0074 when Coverplate was Removed, Showing Mounding up of Material.



Figure A12. Appearance of Target 1-0074 when Loose Debris was Removed.

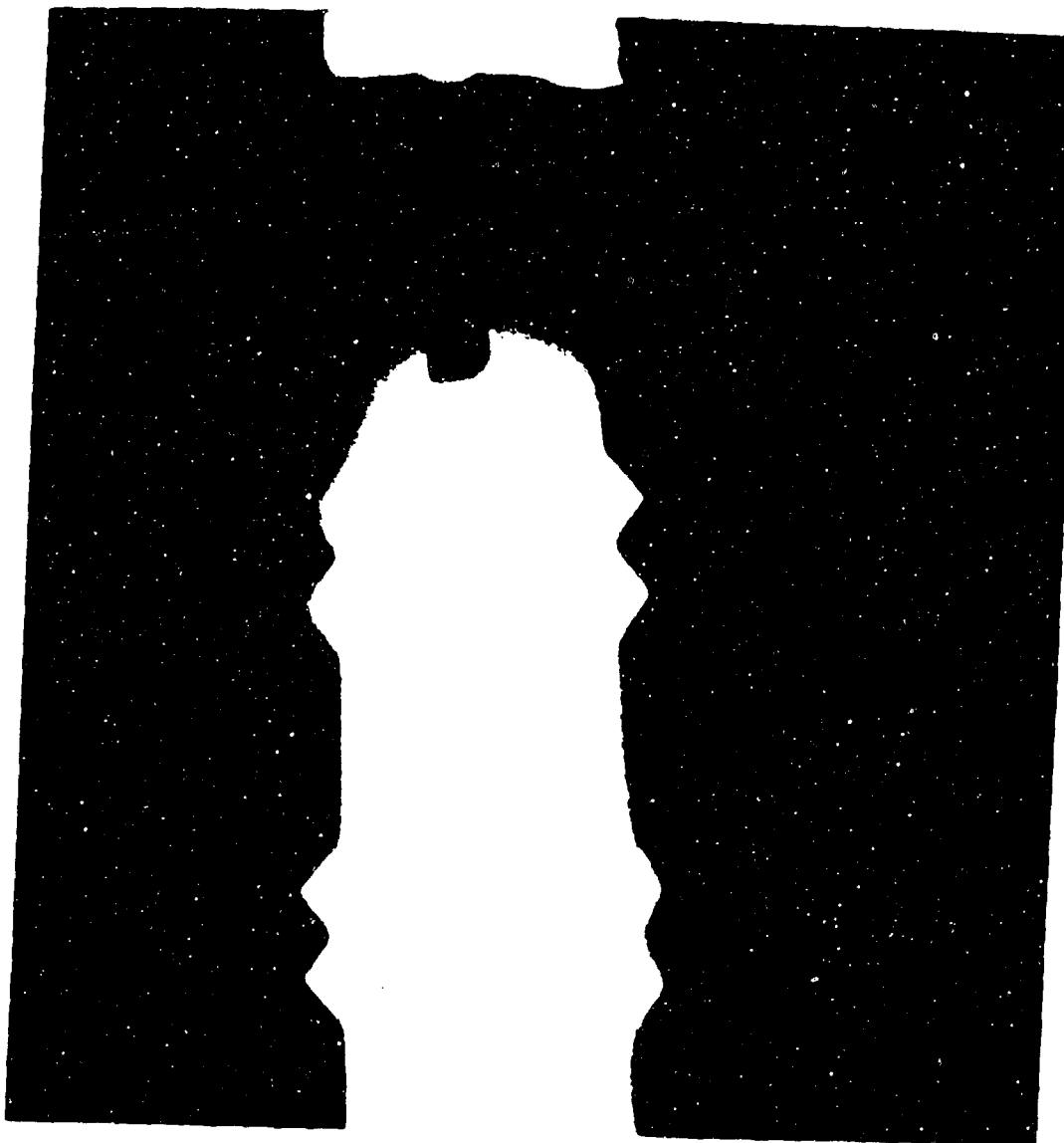


Figure A13. Radiograph of Target 1-0070. (Fiducial Markers are 2 inches Apart and Located on Either Face of the X-rayed Cross Section.)

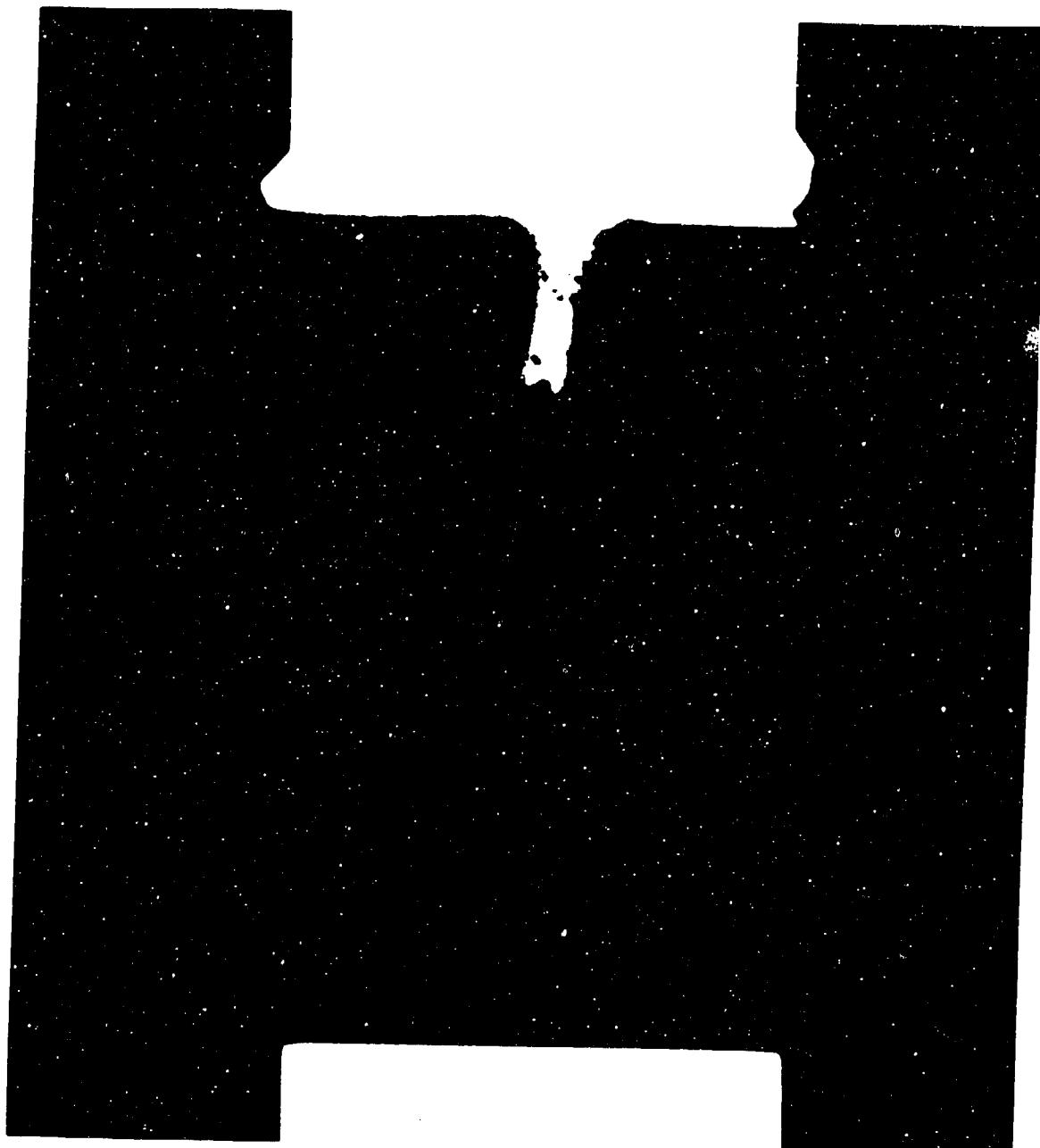


Figure A14. Radiograph of Target 1-0071.



Figure A15. Radiograph of Target 1-0074.

APPENDIX C

Corning Incorporated Facilities and Armor Experience

FACILITIES

Corning offers the customer rapid response through in-place facilities and state-of-the-art equipment.

Sullivan Park

Formed in 1908, one of the first industrial research organizations in the United States, Corning's laboratories are today a major inorganic materials research center. Committed to quality, innovation, and responsiveness to its customers, the laboratory was ranked among the nation's top research laboratories in 1989 by Ernst and Young, an international management consulting firm.

Corning's research and development laboratories, pilot plants, and supporting services are located at Sullivan Park, a 500,000-square-foot research center outside Corning, New York. The facility is devoted to materials and physical sciences, process research and development, and product development. It houses a staff of more than 700, approximately half of whom are scientists and engineers, many with advanced degrees (about 135 hold doctorates).

The support staff can provide the following services:

- chemical and physical properties services
- mathematical and statistical analysis
- process control and instrumentation analysis

- programming and computer services
- technical information services
- an experimental melting and forming plant
- mechanical shop services

Sullivan Park's staff has access to an extensive array of state-of-the-art research equipment and instrumentation needed to extend knowledge, test theories, and refine tentative solutions. These laboratories have also developed many internationally recognized ASTM and ANSI test methods to measure electrical and physical properties of materials. This equipment includes:

- electron beam microprobe
- scanning and transmission electron microscopes
- X-ray diffraction and X-ray fluorescent analyzers
- mass spectrometers
- chromatographs and spectrographs
- image analyzers
- ESCA (Electron Scattering Chemical Analysis)
- SIMS (Secondary Ion Mass Spectrometer)
- various specialized optical waveguide measurement instruments, such as pulsed interferometers and fiber Raman lasers

Sullivan Park is a security-cleared facility in which both classified and unclassified national defense project work is performed.

Process Research Center

The Process Research Center (PRC) is part of Sullivan Park's Research, Development, and Engineering Division. With its large engineering and technical staffs, this "one-stop shop" provides a wide range of process and research and development services including laboratory evaluation, melting, forming, heat treating, oil modeling, and finishing services in addition to its pilot line operations to all Corning divisions, as well as external clients.

Corporate Analytical Services

Corning's scientists are assisted in their work by the Corporate Analytical Services Department, located in Corning, New York. The department utilizes sophisticated equipment and state-of-the-art facilities to solve measurement and analysis problems.

In addition, Corporate Analytical Services provides a state-of-the-art analytical facility capable of handling non-routine problems. One-third of the staff's time is devoted to improving and developing new analytical techniques to ensure valid and useful measurements.

Many different instruments and techniques are available to help in the characterization of materials and processes. These techniques include X-ray crystallography, electron microprobe analysis, electron and optical microscopy, and mass spectrometry.

The newest and most powerful techniques developed for elemental characterization are used, including complexometry, ion-exchange separations, prohydrolysis, combustion analysis, constant current and controlled potential collimator, flame

emission and atomic absorption spectroscopy, and thin-film and gas chromatography. Mechanical, thermal, optical, and electrical properties can be measured over a broad range of parameters and to exceptionally high degrees of precision and accuracy. Microwave measurements are routinely made in-house to 500°C at 1, 3, and 8.5 GHz (X-band).

Computer Services

The Scientific Computer Services Department provides programming and data analysis support for Corning's researchers, scientists, and engineers. Various IBM, DEC (VAX), and other on-line, batch and time-shared computer equipment form a system devoted completely to problems of scientific investigation.

Usually, the programming staff works as part of the research team, helping to organize and instrument experiments so that researchers will get the most amount of useful data, in the form that can be analyzed and handled efficiently.

Such a close liaison between the computer specialists and the researchers means that the computer becomes more than just a routine tool for immediate problem solving. It becomes, instead, a means from which the researcher gains a greater insight into the nature of his problem.

Library Services

An essential part of the research facilities at Corning is the Technical Information Center, a 15,000 volume research library staffed by professional information specialists.

The center subscribes to almost 600 technical and semi-technical periodicals published through-out the world. These

are scanned and abstracted so that articles of interest to Corning's scientists can be brought immediately to their attention. Literature searches are made on request, with translations and loans from outside libraries available.

The center also publishes literature and patent bulletins and newsletters on new developments in the company, Government and international research. It maintains a complete file of all technical reports and papers prepared by the scientific staff.

Canion Plant

Opened in 1966, the 200,000-square-foot Canion, New York, plant is responsible for six separate product areas:

- Fused silica for manned spacecraft windows, high-energy laser optics, and astronomical mirror blanks
- Ultra low expansion (ULE™) glass for lightweight land- and space-based mirrors
- CHEMCOR® frangible glass, including domes for Hellfire and Maverick missiles
- PYROCERAM® radomes for the AMRRAM, Phoenix, Sparrow, and Standard missile programs
- DATA SHIELD® glass tape reels
- MACOR® machinable glass-ceramic materials for applications in the aerospace and nuclear industries, welding, electronics, and medicine

Its unique products have exceptionally high specifications and are subjected to rigid manufacturing and quality control procedures including MIL-Q-9858.

A security-cleared facility, the plant is

staffed by more than 200 highly skilled production and engineering personnel.

Erwin Manufacturing Complex

The Erwin manufacturing complex is comprised of two operational plants, the 200,000-square-foot Electronic Materials Plant and the 250,000-square-foot Ceramics Plant and is staffed by approximately 1,000 diversified, well-trained people.

Opened in 1970, the Electronic Materials Plant has historically been used to develop diversified, new products and take them into successful production operations. Some of its major product lines have included fiber optics, advanced products (including the finishing of missile nose cones), multiform products, quartz crucibles, faceplates, watch crystals, photomask substrates, and military and industrial bulbs.

Today, the major product lines include:

- Specialty ceramics - high-temperature ceramic parts made by slip casting, extruding, and wrapping
- Diesel particulate filters - used to prevent air pollution from diesel engines
- FOTOFORM® - photochemically machinable materials
- Frit (solder glasses) - glass powders for sealing glass-to-glass, glass-to-metals, and glass-to-ceramics
- POLARCOR™ - polarizers of near-infrared radiation
- Thermal protective shielding powder - used to thermally protect spacecraft on reentry

The plant has a slip-casting capability that has produced lithia-alumina-silicate parts up to fifty-five pounds, 15 inches high by 24 inches wide with 1 inch wall thickness. These large parts can also be fired at the Envin Plant.

The wide range of products are subjected to various Quality Control procedures. At present, the plant is incorporating MIL-I-45208A into its Quality Control procedures.

The Ceramics Plant was opened in 1973 to manufacture substrates for catalytic converters and since then has added molten filters and industrial pollution control devices to its high-volume product line.

ARMOR EXPERIENCE

Highly qualified in the materials and development areas, Corning is also experienced in the development of new armor materials.

Corning, a specialty materials manufacturer of high technology inorganic materials, is internationally recognized as a world leader in advanced glass, glass-ceramics, ceramics, and their applications. Heavily committed to research, Corning's Corporate Research and Development Laboratory, established in 1908, is recognized today as the world's foremost research center devoted to fundamental and applied studies of advanced glass and glass-ceramic technologies. Its distinguished history includes the discovery and first patent issued (1960) for glass ceramics to Corning's Dr. S. Donald Stookey. Since this initial discovery, Corning has accumulated the world's most comprehensive body of knowledge on this family of materials.

Corning's advanced material experience includes the development of new armor materials:

- Corning invented and patented (1960s) the composition and process for making B₄C-SiC-Si armor via fusion casting under a Government contract.
- Corning's fiber-reinforced ceramic matrix composites with a bonded hard face of SiC have been tested in ballistic tests at Lawrence

Livermore National Laboratory with very successful results.

- Ballistic evaluations of Corning's unique glass, glass-ceramic, and ceramic materials continue at the US Army's Materials Technology Laboratory (Watertown Arsenal) and the Ballistics Research Laboratory (Aberdeen Proving Ground), Dow Chemical Company, and the University of Dayton Research Institute.
- Corning was awarded a contract by the US Army Research Office under the Balanced Technology Initiative to provide samples of reaction hot pressed materials, toughened alumina, and fiber-reinforced ceramic matrix composites for ballistic testing.

In the last ten years, Corning has done extensive research in the advanced ceramic materials area including glass-ceramics, toughened zirconia and alumina; novel processing of borides, carbides, and nitrides; fine particle powder synthesis; new green forming techniques; and the development of fiber-reinforced ceramic matrix composite materials.

Additionally, Corning has developed the analytical capabilities needed for appropriate characterization of these new materials and processes for correlation of material properties and function.